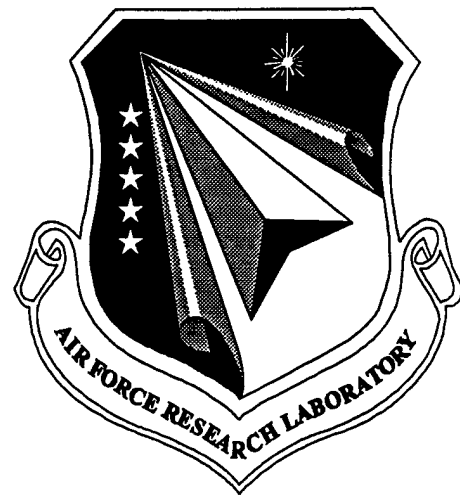


**AFRL-ML-WP-TR-1999-4141**



**GROWTH AND CHARACTERIZATION OF  
DOPED CdSSe AND CdSeTe FOR  
OPTO-ELECTRONIC APPLICATIONS**

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**JULY 1999**

**FINAL REPORT FOR 02/01/1996 – 02/01/1999**

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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE JULY 1999	3. REPORT TYPE AND DATES COVERED FINAL REPORT FOR 02/01/1996 - 02/01/1999	
4. TITLE AND SUBTITLE GROWTH AND CHARACTERIZATION OF DOPED CdSSe AND CdSeTe FOR OPTO-ELECTRONIC APPLICATIONS			5. FUNDING NUMBERS C F33615-96-C-5451 PE 62102 PR 4348 TA 74 WU 03	
6. AUTHOR(S) ARNOLD BURGER STEVEN H. MORGAN				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) FISK UNIVERSITY 1000 SEVENTEENTH AVENUE, N NASHVILLE, TN 37208-3501			8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) MATERIALS AND MANUFACTURING DIRECTORATE AIR FORCE RESEARCH LABORATORY AIR FORCE MATERIEL COMMAND WRIGHT-PATTERSON AFB, OH 45433-7750 POC: MELVIN C. OHMER, AFRL/MLPO, 937-255-4474 EXT. 3233			10. SPONSORING/MONITORING AGENCY REPORT NUMBER  AFRL-ML-WP-TR-1999-4141	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION AVAILABILITY STATEMENT  APPROVED FOR PUBLIC RELEASE, DISTRIBUTION UNLIMITED.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) Control over composition and basic properties of semiconductors is extremely important for the fabrication of efficient and reliable devices. Both macroscopic and microscopic properties of materials strongly depend on their structure. The goal of this three-year project was to establish a collaborative research effort between Fisk University and the Air Force Research Laboratory, aimed at the preparation and optical characterization of doped CdSSe and CdSeTe for their use in opto-electronic applications. The approach involved the preparation of novel crystals followed by investigation of the basic optical and electro-optical properties of the Cd-based ternary compounds. Optical properties were characterized using UV-visible-NIR spectroscopy and infrared spectroscopy, and photorefractive properties were measured using conventional wave-mixing techniques. Defect and transport properties were characterized by photoluminescence and optical absorbance spectroscopies. The achievements under this program led to improvements in the understanding of interactions between material defect properties of II-VI semiconductor crystals and the photorefractive response and other optical properties. This understanding will allow tailoring these defect properties in order to optimize the material for specific applications.				
14. SUBJECT TERMS Cadmium Sulfide, CdS, Cadmium Selenide, CdS, Vanadium V, Vanadium doping, Photorefractive, Infrared Nonlinear Optical			15. NUMBER OF PAGES 10	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT SAR	

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## ABSTRACT

Control over composition and basic properties of semiconductors is extremely important for the fabrication of efficient and reliable devices. Both macroscopic and microscopic properties of materials strongly depend on their structure. The goal of this three-year project was to establish a collaborative research effort between Fisk University and the AF-Wright Laboratory, aimed at the preparation and optical characterization of doped CdSSe and CdSeTe for their use in opto-electronic applications. The approach involved the preparation of novel crystals followed by investigation of the basic optical and electro-optical properties of the Cd-based ternary compounds. Optical properties were characterized using UV-visible-NIR spectroscopy and infrared spectroscopy, and photorefractive properties were measured using conventional wave-mixing techniques. Defect and transport properties were characterized by photoluminescence and optical absorbance spectroscopies. The achievements under this program led to improvements in the understanding of interactions between material defect properties of II-VI semiconductor crystals and the photorefractive response and other optical properties. This understanding will allow tailoring these defect properties in order to optimize the material for specific applications.

## INTRODUCTION

Photorefractive materials are nonlinear optical materials which experience a significant change in the refractive index when exposed to inhomogeneous illumination. These materials have potential device applications in optical signal processing and related areas. These applications include, among others, reversible holographic storage, tracking filters, optical interconnects, and neural net. Although most of the work on photorefractive materials has historically been on oxides such as LiNbO<sub>3</sub> and BaTiO<sub>3</sub> in the visible region of the spectrum, there has been recent interest in II-VI semiconductors for use in the 0.7  $\mu\text{m}$  to 1.5  $\mu\text{m}$  spectral region. This region spans the range of currently available laser diodes, and also includes the telecommunications windows at 1.3  $\mu\text{m}$  and 1.5  $\mu\text{m}$ , as well as the eye-safe region near 1.5  $\mu\text{m}$ . II-VI semiconductors have already been shown to have good figures of merit for device applications in the near infrared. CdTe:V for example, was shown to have a gain coefficient at 1.06  $\mu\text{m}$  more than twice that of GaAs, while ZnTe:V was found to exhibit great potential as a fast, sensitive photorefractive material in the 0.633  $\mu\text{m}$  to 1.32  $\mu\text{m}$  range. The majority of work on the crystalline perfection and electronic properties of II-VI materials, including research done at Fisk, has been done with the aim of optimizing them for use as x-ray and  $\gamma$ -ray detectors, and as substrates for epitaxial growth. There has been little work done on optimizing these materials for photorefractive applications, or relating photorefractive properties to specific material defect properties. Although photorefractive studies on II-VI's have to date been very promising, there is still the need for improvement in materials and crystal growth in order to obtain sizable crystals

with controllable defect densities.

## METHODS, ASSUMPTIONS AND PROCEDURE

Our goal in this three-year project was to undertake a thorough study of the relationship between the defect properties, optical and electronic properties, and photorefractive response of II-VI semiconductors. Specific objectives are (1) to determine the effect of purification, crystal growth techniques and post-growth treatments on trap density, carrier transport properties, absorption coefficient and other properties relevant to the material's photorefractive performance, (2) to use selected transition metals as dopants in order to evaluate the relationship between dopant species and concentration, defect and transport properties, and photorefractive properties, (3) to measure the time response of the photorefractive effect and its dependence on defect properties, and (4) to study the effectiveness of adjusting the bandgap and other properties by alloying in order to optimize the material for use at a specific wavelength. One major issue in this investigation will be to establish the relation between free and bound carrier effects in determining the magnitude and response time of the optical changes that occur. The relationship between materials properties and photorefractive response can be illustrated using a simple band transport model with a single defect [Kuhktarev 1979]. The total defect concentration is  $N_T$ , the concentration of ionized defects is  $N_0$ , and the concentration of neutral defects is  $(N_T - N_0)$ .

Thermal and optical emission of electrons and holes from the defect are governed by  $\beta$  and  $S$  respectively, while the recombination coefficients are given by  $\gamma_n$  and  $\gamma_p$ . In a two-beam coupling experiment, with no applied field and incident intensity  $I_0$ , the space-charge field is described by

$$E_1 = -im \frac{k_B T}{e} \frac{k}{1 + (k^2/k_0^2)} \xi_0 \quad (1)$$

where  $k$  is the grating wavevector,  $k_0 = [(e^2/\epsilon k_B T)N_{\text{eff}}]^{1/2}$  is the inverse of the Debye screening length,  $N_{\text{eff}} = N_0/N_T(N_T - N_0)$  is the effective trap density and  $\xi_0$  is the electron-hole competition factor.

The photorefractive gain can be expressed as:

$$\Gamma = \frac{2\pi n_0^3 r_{\text{eff}}}{\lambda} \frac{k_B T}{e} \frac{k}{1 + (k^2/k_0^2)} \xi \quad (2)$$

An important intrinsic property which affects the photorefractive response of a given material is the product  $n_0^3 r_{\text{eff}}$ , where  $n_0$  is the linear refractive index, and  $r_{\text{eff}}$  is the appropriate component of the electro-optic coefficient.

The reasons for studying ternary compounds come from some limitations existent in the binary compounds. For example, one limitation in the development of CdTe is the propensity of this material to include during growth a large amount of extensive defects, such as grains and twinning. For CdSe, one of the end members of these ternary systems, growth of large crystals is easier and shows no twins or grains. CdSe is a promising photorefractive material for other reasons as well, although its  $n^3 r_{\text{eff}}$  value is lower than that of CdTe. CdSe exhibits a large photoconductivity over a wider range than CdTe, and also is obtainable with higher resistivity (~2 orders of magnitude) than CdTe. Finally, the existence of deep trap levels suggests efficient photorefractivity. CdS is the end member with the largest bandgap thus allowing the possibility of preparing mixed crystals with transparency extending into the visible spectrum, and adjusting the bandgap and other properties in order to optimize the material for use at a specific wavelength. Vanadium doping has previously been shown to result in high photorefractive sensitivity for both CdTe and ZnTe [Bylsma 1987, von Bardeleben 1993, Schwartz 1994, Belaud 1994, Launay 1992, Moisan 1994, Partovi 1990, Rzepka 1994, Ziari

1992], and is a natural starting point for dopant studies in the proposed ternary systems.

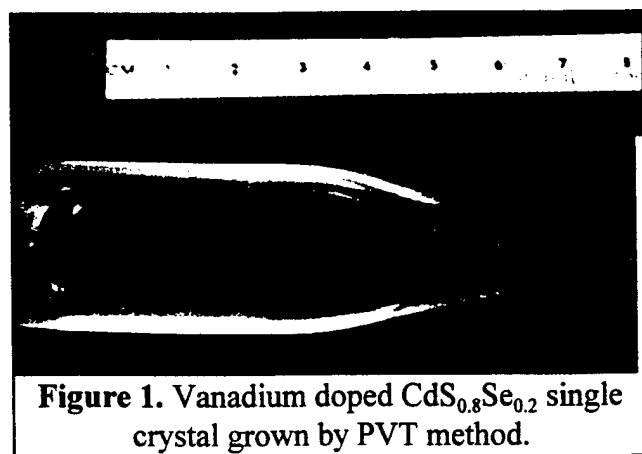
Values of  $n^3 r_{\text{eff}}$  for various II-VI crystals and GaAs are listed in Table I, along with values for band-gap and dark conductivity.

**TABLE 1** Bandgap, dark conductivity, index of refraction and effective electrooptic coefficient for selected II-VI semiconductors and gallium arsenide [Kaminow 1986]

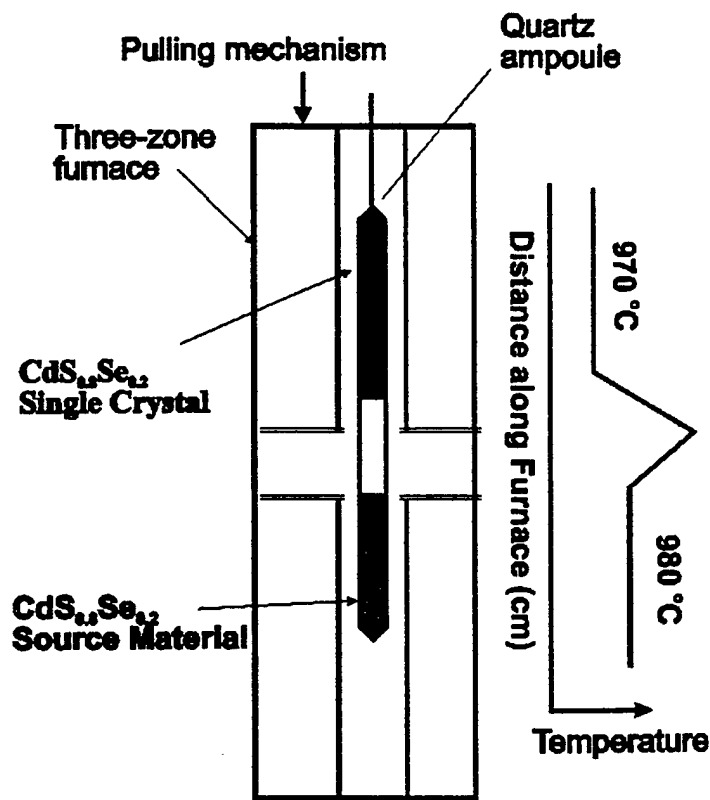
Material	$E_g$ (eV)	Crystal Class	$\sigma_d$ ( $\Omega$ -cm)	$n_0$ ( $\lambda$ in $\mu\text{m}$ )	$r_{\text{eff}}$ (pm/V) ( $\lambda$ in $\mu\text{m}$ )	$n_0^3 r_{\text{eff}}$ (pm/V)
CdTe	1.5	43m	$<10^{-10}$	2.8	5.5	120
CdSe	1.7	6mm	$<10^{-12}$	2.5(3.39)	1.8(3.39)	28
ZnTe	2.3	43m	$<10^{-9}$	2.9(0.70) 2.7(2.06)	4.3(0.63) 3.2(3.39)	105
CdS	2.4	6mm	$<10^{-10}$	2.5(0.63)	1.1(0.63)	17
ZnSe	2.7	43m	$<10^{-12}$	2.7(0.55)	2.0(0.63)	39
GaAs	1.4	43m	$<10^{-10}$	3.5(1.02)	1.3(1.06)	57

## RESULTS AND DISCUSSION

Undoped and vanadium doped  $\text{CdS}_{0.8}\text{Se}_{0.2}$  single crystals were successfully grown by PVT. Both crystals were all 1.1 cm in diameter and about 6.0 cm in length. Figure 1 shows the as-grown vanadium doped crystal while still in the quartz growth ampule. The electrical resistivity was measured and was in the range of  $10^7 \Omega$ -cm.



**Figure 1.** Vanadium doped  $\text{CdS}_{0.8}\text{Se}_{0.2}$  single crystal grown by PVT method.



### Physical Vapor Transport (PVT)

Figure 2. Experimental set up for crystal growth using the Physical Vapor Transport.

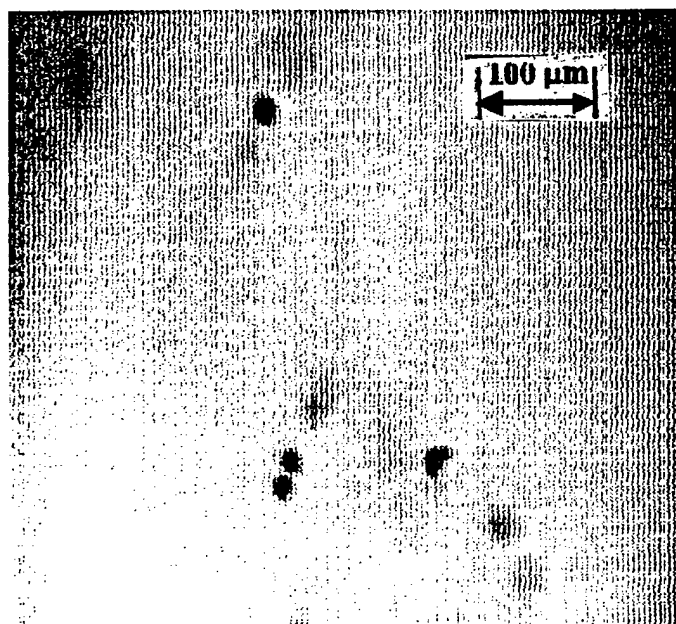


Figure 3. Optical microscopy image of an undoped CdSSe crystal showing the presence of precipitates.

voltages up to 100 V. A rectangular  $8 \times 5 \times 4 \text{ mm}^3$  parallelepiped sample with the orientation of  $c$ -axis along 8 mm edge was cut and polished. Photorefractive two-beam coupling measurement show that the gain is comparable to the best value from other semiconductor materials [Morgan 87].

Spectroscopic measurements were obtained on both characterization of doped and undoped CdSSe samples. A photorefractivity measurement system was designed, built and used in the measurement of photorefractive properties. Figure 4. shows the PL spectra of undoped and vanadium doped  $\text{CdS}_{0.8}\text{Se}_{0.2}$  crystals.

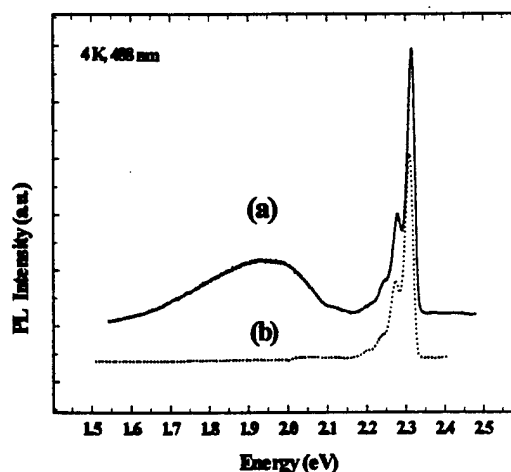


Figure 4. Low temperature photoluminescence spectra for vanadium doped (a) and undoped CdSSe single crystals

For the undoped crystal a strong bound exciton peak is located at 2.307 eV with two phonon replicas at 2.273 and 2.241 eV. The spacing between the phonon replicas is about 32 meV, which is in between the values of the longitudinal (LO) phonon of 38 meV in CdS and the LO phonon of 24 meV in CdSe [Arora 87], respectively. The vanadium doped crystal shows the similar strong band at 2.313 eV along with two phonon replicas at 2.279 and 2.244 with 35 meV spacing. Additional broad emission band from 1.7 to 2.1 eV (centered at 1.95 eV) is shown only for vanadium doped crystal, and we attributed this to vanadium. Similar emission band was observed in a vanadium-doped CdTe [Arora 87]. Low temperature (16 and 25 K) and room temperature IR transmittance spectra on doped crystal all shows a broad absorption band between  $6,500$  and  $10,500 \text{ cm}^{-1}$  (0.8-1.3 eV), and we

attributed this absorption band to vanadium dopant. In the area of synthesis and crystal growth the tasks were to purify starting materials, determine growth conditions and grow first vanadium doped and undoped CdSSe crystals, followed by wafer sectioning, polishing and etching. Several upgrades were accomplished in the crystal growth area. The installation of the new crystal growth system including (three zone oven, operation up to 1200 °C) was completed. The system was characterized (thermal profile, achievable temperature gradients and thermal stability) and is ready for new growth runs within in the renewal project. A two zone transparent oven was also assembled. A fully computerized zone refiner was constructed.

Figure 3 above shows the optical microscopy images of etched surfaces in reflective mode and transmission images into undoped bulk crystal. The shape of the etch pit is elongate and irregular which is similar to the etch pits on CdS using the same etchant [Su 90]. The EPD for undoped and vanadium doped crystals are  $5 \times 10^4$  and  $7 \times 10^4$  per  $\text{cm}^2$ , respectively. The length of the etch pits ranges from 5 - 70  $\mu\text{m}$ . Second phase precipitates/inclusions were observed inside both doped and undoped crystals. The size and concentration are 1 - 3  $\mu\text{m}$  and  $10^3$  -  $10^4$  / $\text{cm}^2$ . In a previous differential scanning calorimetry (DSC) study on PVT grown ZnSe crystals [Chen 95], Se precipitates were observed and distributed uniformly through bulk crystal. The concentration of Se precipitates was estimated to be about  $10^{13}/\text{cm}^2$ , and was converted to 0.8 wt%. Therefore we speculate these second phase may be rich in group VI solid solution. Further composition identification is needed. The distribution of these second phase is of great interests and may link to gravity direction. Micrographs were taken along growth direction which is parallel with gravity direction. The distribution of second phase is not uniform on the same surface. They distribute in a clockwise direction into bulk crystal along growth direction. This distribution behavior may caused by convection effect induced by gravity during crystal growth.

#### Photorefractive measurements

Figure 5 below describes the two beam coupling experiment.

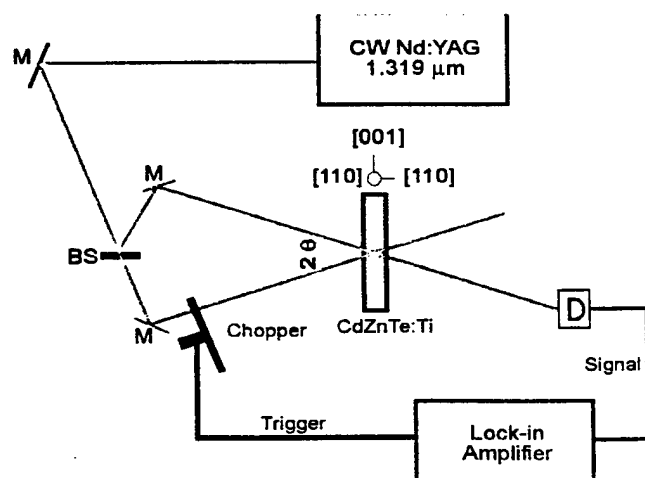


Figure 5. Two beam coupling experiment for the measurement of the photorefractive effect

A large photorefractive gain coefficient of  $0.24 \text{ cm}^{-1}$  was observed at 633 nm with an optical intensity of  $60 \text{ mW}/\text{cm}^2$  and a grating period of 1.6  $\mu\text{m}$ . At this wavelength, we measured the photorefractive gain as a function of the grating period and incident optical intensity. To our knowledge, this is the first observation of the photorefractive effect in vanadium doped CdSSe crystals. The observed photorefractive sensitivity is significant among photorefractive materials operating in the visible range. The grown crystal has a large crystal size, a good optical quality, and a medium resistivity of  $10^6$  -  $10^8 \Omega\text{-cm}$ . With a significant photorefractive effect, the CdSSe:V crystals are promising for many device applications based on photorefractive effect, including optical limiting devices in the visible region.

#### Deliverables

Purification of approx. 75 g of CdSe and 75 g of CdS, a new crystal growth oven and controller were installed. The following crystals were grown: 4 crystals of  $\text{Te}_x\text{Se}_{1-x}$  (high purity elements,  $x=0, 0.1, 0.2$ ) by-product of our purification effort, were prepared and shipped to MLPO/MLPJ, 4 CdSSe crystal growth experiments were performed, 3 were successful and resulted in large vanadium doped crystals. The CdSSe crystals were grown by physical vapor transport (PVT) and doped with 150 ppm vanadium for creating trap centers. One crystal was grown undoped.

Additional crystals grown and delivered to AFRL were: 3 crystals of Cr:CdSe, 1 preliminary crystal of  $\text{AgGaTe}_2$ . The starting material was made of the components  $\text{Ag}_2\text{Te}$  and  $\text{Ga}_2\text{Te}_3$ , their melting point are 950 °C and 810 °C respectively. The technique used is designed to avoid the explosions we have had during the previous two experiments, the above components, in stoichiometric weights, were placed in a quartz ampoule and were baked at 700 °C in a dynamic vacuum for 24 hours, to remove water from them.

#### Collaborations

A wafer of the doped CdSSe crystal was cut in the shape of a 30° crystallographically oriented prism, polished and shipped to for the determination of the birefringence in this crystal at WL; an oriented sample was cut and shipped to Dr. Larry Halliburton at WVU, for epr measurements. UV-Vis and mid-IR spectra were taken and the crystals were delivered to WL for additional low temperature characterization by Dr. M. Ohmer, Dr. J. Goldstein, Dr. S. Guha, Dr. J. McKay and Dr. K. Schepler, Dr. P. Hood and Dr. G. Brown of AFRL.

#### Student training:

Five graduate (M.S. degree) students have participated in the projects. At present, one of the students participants, Ms. Michelle Davis, is pursuing a Ph.D. degree at Ohio State University.



Several undergraduates were involved in the research through the Summer Research Program at Fisk

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## **APPENDIX: Publications and Presentations related to the project**

### **Publications:**

S. H. Morgan, Zhengda Pan, Michelle Davis, Kuo-Tong Chen, Henry Chen, Swanson L. Davis, and Arnold Burger, "Photorefractive Effect in CdSSe:V Crystals", Proceedings of the MRS 1997 Spring Meeting, Optical Limiting Devices (Invited Paper), Vol. 479 (1997) 209

Kuo-Tong Chen, Michelle Davis, Steven Morgan and Arnold Burger, Crystal Growth And "Characterization of Vanadium Doped And Undoped CdSSe", SPIE Proceedings of Spacecraft Instrument Program and Materials Research in Low Gravity, Vol. 3132 (1997) 58

K. Chattopadhyay, S. Feth, H. Chen, A. Burger, Ching-Hua Su, "Characterization of Semi-insulating CdTe Crystals Grown by Horizontal Seeded Physical Vapor Transport", J. Cryst. Growth, 191, (1998) 377-385.

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K. Chattopadhyay, K. M. Pour, S. U. Egarievwe, J. O. Nday, H. Chen, X. Ma, S. Morgan, and Arnold Burger, "Study of CdSSe:V and CdMnTe:V Photorefractive Effect", accepted for publication in Journal of Electronic Materials, 1999.

### **Presentations:**

M. Davis, L. Collins, K. Dyer, J. Tong, A. Ueda, H. Chen, K.-T. Chen, A. Burger, Z. Pan and S. H. Morgan, "Photorefractivity In A Titanium Doped ZnCdTe Crystal", NASA University Research Centers First National Students= Conference, North Carolina A&T State University, Greensboro, NC, Mar. 31-Apr. 2, 1996

W. Palosz, M. A. George, W. E. Collins, K. -T. Chen, Y. Zhang, Z. Hu and A. Burger, "Growth and Characterization of Cadmium Zinc Telluride Crystals Grown by Seeded Physical Vapor Deposition", The Tenth American Conference on Crystal Growth in conjunction with The Ninth International Conference on Vapor Phase and Epitaxy, August 4-9, 1996 Vail, Colorado.

Michelle Davis, Zhangda Pan, Kuo-Tong Chen, Henry Chen, Swanson L. Davis, Arnold Burger and Steven Morgan, "Photorefractive Effect in a Vanadium Doped CdSSe Crystal", NASA-URC Technical Conference on Education, Aeronautics, Space Autonomy, Earth, and Environment, Feb. 16-19, 1997, Albuquerque, NM

Kuo-Tong Chen, Detang Shi, S. H. Morgan, W. Eugene Collins and Arnold Burger, "Growth of Bulk Wide Bandgap Semiconductor Crystals And Their Potential Applications", NASA-URC Technical Conference on Education, Aeronautics, Space, Autonomy, Earth, and Environment, Feb. 16-19, 1997, Albuquerque, NM

Shekhar Guha, Patrick J. Hood, Melvin C. Ohmer, Kuo-Tong Chen, Steve Morgan and Arnold

Burger, Fisk University, "Infrared optical limiting Properties of Mercury Cadmium Telluride and Tellurium Selenide", The 1997 IRIS Specialty Group Meeting on Infrared Countermeasures at the Applied Physics Laboratory, Laurel, MD

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